

MORPHOLOGY EVALUATION OF SPUTTERED Au FILMS ONTO MICA BY ATOMIC FORCE MICROSCOPY

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RESUMO

A microscopia de força atômica (AFM) é empregada na caracterização da morfologia de filme de Au depositado por sputtering sobre mica clivada, em condições típicas de preparação de amostras para MEV. Os resultados tem como base de análise a variação de rugosidade média quadrática (RMS) com respeito à superfície anterior à formação do filme. Os resultados indicam uma deposição com relativa homogeneidade e com formação de núcleos nas dimensões finais em torno de 40 nm de altura por 30 nm de diâmetro. O aumento de área provocada pelo depósito, quando medida por AFM, é encontrado como sendo em torno de 5,5 vezes.

ABSTRACT

Atomic force microscope (AFM) was used to investigate the surface morphology of Au sputtered onto cleaved mica in ordinary deposition as used to SEM analysis. The data have as basis the variation of the surface morphology by the film formation and the RMS roughness assessment. The results indicate a relative homogeneity in the deposited film with nuclei formation of dimensions around 40 nm height by 30 nm diameters. The overall area, when measured by AFM way, is found to be approximately 5.5 times large due to deposition.

INTRODUCTION

The sputtering process allowed a relatively easy and quick formation of metallic films, being a much simpler and faster than other deposition techniques. Due to this characteristics, the sputtering is extensively employed for metallic coatings onto non-conductive materials as ceramics and polymers at relatively lower temperatures. The morphology characteristics of the sputtered films have been recently subject of some works¹⁻³, where the

experimental parameters are attributed to have direct influence on the final film morphology.

For SEM (scanning electron microscopy) analysis, the sputtering technique is widespread used, where the morphological condition of the deposited metal is assumed to make no significant difference since the incident beam penetrates into the whole film thickness turning it invisible to SEM imaging. However, in applications as thin films sensors or catalytic electrodes, where sputtered coating is also used, the features of the film plays an important role. An increasing or even a reduction of the roughness in these devices may bring about an undesirable result. That is also decisive in wetting conditions and direct interferes in the SEM backscattered electrons profile⁴⁻⁶.

In this work we are interested in evaluate alterations in surface structure performed by a conventional film of Au sputtered on materials for SEM imaging, where cleaved mica is used to compare changes in surface topography.

EXPERIMENTAL

The depositions were carried out in a small-sized sputter-coating apparatus, from Balzer, onto sheets of natural mica, in room temperature, cleaved to ~ 0.1 mm thick and clamped against the cathode located straight opposite of the Au source. The electrodes were laid at a distance of 35 mm, under argon flow, keeping a pressure on the order of 0.07 mbar in the chamber. The sputtering operation time was 130 seconds with working current of 15mA. The gold film sputtered in these experimental conditions has typical thickness between 30 and 50 nm.

The surfaces, prior and after the Au deposition, were AFM (atomic force microscopy) scanned by using a TopoMetrix (Discover TMX 2010) system. The morphology and topographical features were analyzed by software TOPOSPM v. 3.06.06. All images were generated in contact mode. A tip specially devised for roughness surface, commercially named *supertip*, (mod. 1700 - Topometrix) was used.

RESULTS AND DISCUSSION

Figure 1 shows a typical sample of AFM image of cleaved clean mica (A) ($1\mu\text{m} \times 1\mu\text{m}$) and the same surface after Au deposition (B). The AFM scanning provides nanometric scaled images where the smoothness of the surfaces can be visually assessed. The maximum graduation scale (on the left) concerns the higher point detected on the surface.

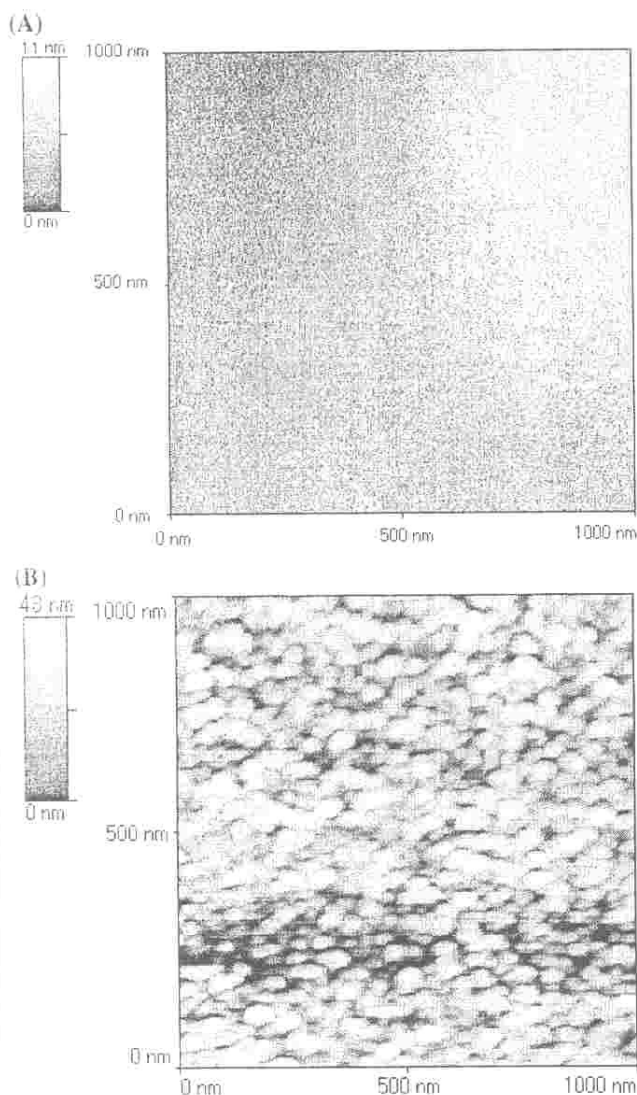


Figure 1 – Atomic force microscopy visual aspect of surfaces: (A) Clean like, cleaved mica; (B) After gold deposition.

The figures essentially inform that the morphology of the observed surface is greatly changed by the sputtering process. In the Au deposited surface, the final mass distribution of sputtered clusters characterizes the film morphology. It is worth to be stressed that the AFM images are predominately composed by convolution effects due to tip-surface interactions. Nevertheless, the main aim of this work is to compare changing on the

roughness performed by the sputtering deposition process. The presence of various grains/aggregates on substrate suggests that the deposition of Au proceed by formation of individual nuclei and subsequent growth resulting in films with a corrugated final morphology.

The AFM resident software allows the drawing of an isolated line of the composed images, which is shown in Figure 2. These cross sections present the variation of the surface height taken along the scanned samples. By comparing both of the profiles it is possible to appraise the elevation of surface corrugation due to the deposition is quite significant. Here, also the inclination of the drawn hills corresponds to artifacts due to tip's faces, which must be taken into consideration. Since the experimental analysis is the same to coated and non-coated mica, is important to put both lines together for a simple comparison.

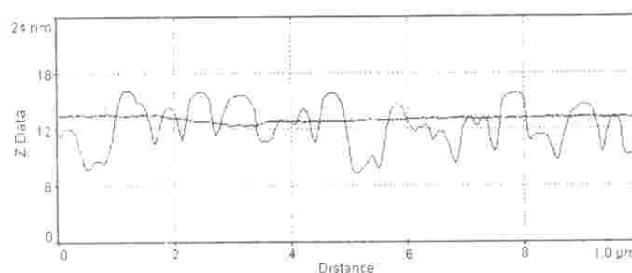


Figure 2 – Two random profile lines took from the AFM scanning. Flat like: cleaved mica; Hill-like structure: profile of the Au deposited film.

By numerical analysis of the scanned areas, it is found a numerical height variation of 9.16 nm to cleaved mica and 43.75 nm to the deposited surface, what regarding the scale of the measurement, it means a relative homogeneity of the deposited layer. The roughness amplitude of the Au features is similar to those measured by Porath and co-authors by STM². That is important to be noticed that the amplitude (Z ranges) is established by the measurement of the maximum distance between the minor to the higher irregularities on the surface. Along the deposited material, the distances are taken between the tops of the nuclei, not directly from the substrate as exemplified in Figure 3.

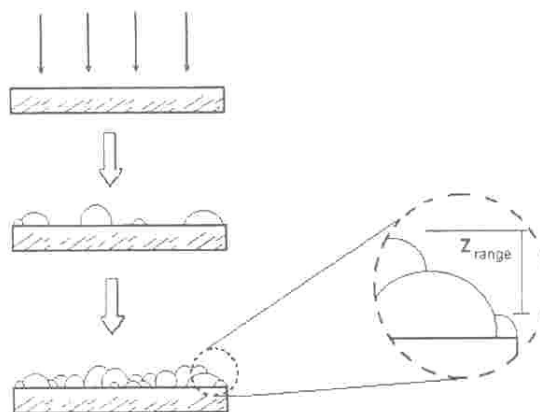


Figure 3 – Illustration of the maximum Z range measured by the AFM in the final coated surface.

In Figure 4 is showed the tridimensional projection of a zoomed central region illustrating the nucleated cluster aspect. Evidently to considered the actual nuclei forms we have to isolate the image artifacts inherent of the AFM scanning system, which in images of individuals nucleus predominate the convolution effects⁷⁻⁹, as motioned before. Despite generating artifacts, the AFM is a powerful tool for characterizing surface structure of which images allow important morphological interpretations.

As already presented, if we considered the corrugation values, and the nuclei average height, there is a relative homogeneity of the deposition, being the mica substrate complete covered by the Au film. The typical diameter of the final Au clusters can be thus roughly estimated from the profile lines, as probably presenting rounded like shapes, and dimensions of the order of 30 nm wide by 40 nm high. In spite of all limitation of the AFM system, these dimensions are in concordance to the size of Au grains nucleation, as theoretically predicted by Zinke-Altmang¹⁰, and microscopically measured by Porath².

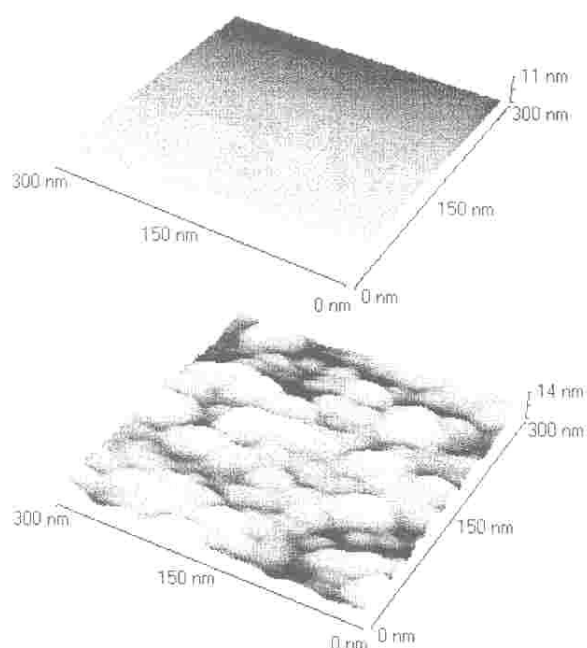


Figure 4 – 3D projection of the clean mica and the gold nuclei (convolution effects predominates in the image).

For an overall quantitative analysis we have computed the arithmetic roughness (R_a) along a single line located at a central region, and the RMS roughness over square areas of various sizes in the scanned samples. These are the most common statistical descriptors for surface analysis, being the basic relations and methodology of calculation previously described¹¹.

The data are presented in the table 1. An area increase of approximately 5.5 times is found in consequence of the deposition. This increase of area is also close to that got by non-direct measurements, as electrochemical via, in tests performed on Au sputtered on ceramic samples^{12,13}.

Table 1 – Numerical average values of the roughness

	Mica	Au film
Roughness R_a (Cross-section)	0.15 nm	1.73 nm
Roughness RMS (area)	1.16 nm	6.26 nm

From the dimensions values it is possible to calculate qualitatively the roughness factor, R_s , by considering the clusters as having cone forms with circular base radii of 30 nm and height of 40nm, being:

$$R_s = \frac{A_R}{A_0} \quad (1)$$

where A_R is the surface area and A_0 is the surface projected. The R_s found by the above relationship is 3.4. That means a good concordance with the roughness area increasing as direct obtained by the AFM. From these results are evidently that the deposited sputtering gold in conditions ordinary used to SEM imaging, introduces changes in the surface planarity. These changes could interfere in SEM measurements with high amplification¹⁰ and specially in electrochemistry tests, such as in the evaluation of surface fracture roughness, e.g., in polycrystalline ceramics, when determined by cyclic voltammetry^{12,14}.

CONCLUSIONS

It was found that the Au sputtering deposition in ordinary SEM condition, in spite of its relatively homogeneity, is characterized by forming small-sized clusters. The increase of the area due to deposition, when measured by AFM technique, was found to be in the order of 5 times. For an actual assessment of the nucleus geometry and surface topography, image artifact should be take into consideration. Anyway, the data are close to those attained by other methodologies. The results confirm that depositions by sputtering do introduce changes on the analyzed surface. These films 'invisible' to SEM, and 'observable' by AFM, can interferes in surface imaging and other applications such as sensors or electrochemical measurements.

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